Effective Cross-linking Dyeing Method for Jute Fabric with Reactive Dyes

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Abstract

With a view to develop an effective dyeing method for jute fabric, jute fabric known as Carpet Backing Cloth (CBC), made from jute fiber, was first desized, scoured, and bleached with hydrogen peroxide. Simultaneous dyeing and finishing methods were carried out using various reactive dyes with some cross-linking agents like Fixapret CPN, Fixapret ECOs of BASF, and Indosol E-50 Powder (Sandoz) along with some catalysts ($MgCl_2$, $6H_2O$) and softening agent. Three types of cross-linking methods such as dry, moist, and wet were employed. The cross-linking method was optimized by treatment of bleached CBC with different concentrations of cross-linking agents and catalysts applying pad-dry-cure method at different temperatures. Significant improvement was found in dye fixation rate, dye fastness (washing, rubbing, light and perspiration) properties, and tensile strength of cross-linked dyed jute fabric. Regarding all dyeing and physical properties, Indosol E-50 powder along with various reactive dyes achieved highly satisfactory results. Fixapret CPN may also be comparable to Indosol E-50 powder.

Keywords: Jute fabric, Cross-linking method, Cross-linking agent, Reactive dyes, Fastness Properties

Introduction

Jute is a natural fiber composed of cellulose (58-63%), hemicelluloses (21-24%), lignin (12-14%), wax (0.4-0.8%), pectin (0.2-0.5%), protein (0.8-1.5%), mineral matters (0.6-1.2%), and traces of pigments (Macmillan, 1951; Jabbar et al., 1964; Trivedi & Mehta, 1973; Spurten & Graffin, 1954; Shahidullah, 2005). Jute fiber is a complex mixture of chemical compounds, which are built up by natural process (photosynthesis) during the growth of jute fiber in the plant stem. The composition of jute fiber is not uniform. The conditions of soil, climate, maturity of the plant, retting etc. make considerable variation in the constituents of the fiber. It has been indicated by paper chromatography analysis that hemicelluloses is composed of xylose, arabinose, ramose, galactose, and 4-0 methylglucuronic acid where alpha cellulose is mainly comprised of glucose units together with xylose and arabinose.

Due to inroads of synthetic fibers, the conventional use of jute products is declined. Hence, it needs diversification. This is why it is essential to make the jute fabrics attractive and cost-effective by dyeing and finishing (Kamal, 2005). On successful development of effective cross-linking dyeing method for jute, production of value added colorful jute products will be easier. Also, the demand of jute products will be increase in the world market.

A few studies have been carried out to investigate the cross-linking technique for identifying a tri functional cross-linking agent and a mono functional dye can yield high degree of fixation under suitable condition (Lutzel, 1966). In some cases, jute fiber might lose tensile strength to a great extent due to brittleness of fiber while curing at higher temperature. The cross-linking agents containing two or more reactive groups are capable of forming stable chemical bonds with both the cellulose resulting to high color yield (Lutzel, 1966; Trotman, 1984), and the color will not be washed out after washing. The quality of the fabric will be improved to a great extent resulting to a high degree of dye fixation, high color fastness rating with dimensional stability, and crease recovery.

Simultaneous dyeing and resin finishing of jute fabrics using DMDHEU as cross-linking agent was carried out (Bagchi et al., 1990), and it was assumed that some dye fixation takes place by the covalent bonds between the nucleophilic groups of dyestuffs (NH_2, OH) and the methylol groups of finishing agents. Some of these dyestuffs were entrapped within the resinification product of finishing agent. It was assumed that if the dye contains electron donor group (nH_2, OH), methylol group of DMDHEU forms a covalent bond with $NH_{2,}$ or the OH groups of dyestuff.

In recent years, however, the feasibility of combined dyeing and finishing of cellulose fabrics with various dyes was studied (Shet, 1981; Rattee, 1962; Hashem et al., 1981; Kamel et al., 1990; Kamal et al., 1989; Alfy, 1989).

Lutzel (1966) and Fowler (1964) reported that covalent bond could be established between cellulose and dyes containing mucleophilic groups by employing cross-linking agents, which are able to provide several positively charged atoms.

According to Som et al. (1989), it was observed that the cross-linking treatment of jute with DMDHEU made it less hygroscopic. This means that reduction in hygroscopic property is reused in cross-linking treatment.

Chemical processing of jute such as various pretreatment, dyeing, and cross-linking with DMEU and DMDHEU was studied in order to evaluate the effect of chemical treatment on undyed fabric properties, such as tensile strength, crease recovery, wash and light fastness (Ganguly et al., 1989). There was a great loss of strength when the bleached and causticized fabrics were cross-linked with DMEU along with M_gCl_2 using acetic acid to increase acidic pH.

According to Oh et al. (2001), research was carried out on simultaneous dyeing and anti crease finishing of cotton fabric with reactive dyes and citric acid using pad-dry-cure process. The obtained results indicated that the properties (such as color strength, dry wrinkle recovery angle and breaking strength) of dyed and finished cotton fabrics were affected by a variety of factors. They concluded that relatively satisfactory properties of dyed and finished fabric could be obtained with appropriate adjustment of the treatment conditions.

Research findings of Kamal (2005) revealed that optimum dye fixation rate (70%) could be achieved with the cross-linking agent Fixapret CPN (100 g/l) by curing at 160 ^oC for 2 minutes. On the other hand, highly satisfactory dye fixation rate (94%) could be achieved with the cross-linking agent Indosol E-50 powder (40 g/l) by curing at 140 ^oC for 2 minutes. Fixapret ECOs obtained very low dye fixation rate. This is because it did not cross-link with dye and jute fiber.

From a comparative study done by Kamal (2005) of Dry cross-linking method, Wet cross-linking method, and Moist cross-linking method, it was found that Dry cross-linking method attained the best performance regarding dyeing property and physical properties of dyed fabric in simultaneous dyeing and finishing of jute fabric.

In view of above situation, an attempt was undertaken to carry out research to develop standard and cost-effective cross-linking dyeing method for jute fabric using reactive dyes.

Material and Methods

Carpet backing Cloth (CBC) made from *Corchorus Olitorius* (Bangla Tossa) fiber was first desized, scoured, and bleached with hydrogen peroxide. Simultaneous dyeing and finishing methods were carried out using various reactive dyes with some cross-linking agents such as Fixapret CPN (DMDHEU) of BASF and Indosol E-50 Powder (Sandoz) along with some catalysts ($MgCl_2$, $6H_2O$) and softening agent. Dry cross-linking method was employed.

Cross-linking Agents used with Reactive Dyes

DMDHEU, commonly known as Fixapret CPN, Fixapret ECOs and Indosol E-50 powder (Santos), were selected and used as cross-linking agents together with reactive dyes.

Reactive Dyes used for the Study

Seven varieties of portion M-types of reactive dyes for simultaneous dyeing of jute fabric were selected. The names of the dyes are as follows: Procion Yellow MX4R, Procion Red MX8B, Procion Orange MX2R, Procion Brown MXGRN, Procion Blue MX2R, Procion Blue MX2R, Procion Yellow MX3G and Procion Turquoise MXG.

Optimization of Dye Fixation Condition

Kamal (2005) has optimized the dye fixation condition. Three types of cross-linking agents namely Fixapret CPN (DMDHEU, BASF), Fixapret ECOs (DMDHEU, BASF), and Indosol E-50 powder (Sandoz) were selected and used together with Portion M-types of reactive dyes for simultaneous dyeing of jute fabrics. The concentration of the cross-linking agent were 80-120 g/l (for DMDHEU i.e., Fixapret CPN & Fixapret ECOs) and 15-50 g/l (for Indosol E-powder) respectively. Research findings revealed that optimum dye fixation rate (70%) could be achieved with the cross-linking agent Fixapret CPN (100 g/l) by curing at 160 ^{o}C for 2 minutes. On the other hand, highly satisfactory dye fixation rate (94%) could be achieved with the cross-linking agent Indosol E-50 powder (40 g/l) by curing at 140 ^{o}C for 2 minutes.
Fixapret Ecos obtained very low dye fixation rate. This is because it did not cross-link with dye and jute fiber. Hence, Fixapret Ecos was not used in the final study.

Omitting Fixapret ECOs

The performance of Fixapret ECOs in attaining dye fixation rate was observed, and it turned out to be very poor during optimization. Hence, Fixapret ECOs was not used in the final study.

Selection of Cross-linking Method

Kamal (2005) had also done a comparative study of Dry cross-linking method, Wet cross-linking method, and Moist cross-linking method. It was found that Dry cross-linking method attained the best performance regarding dyeing property and physical properties of dyed fabric in simultaneous dyeing and finishing of jute fabric. Therefore, Dry cross-linking method was finally selected for the study.

Dry Cross-linking Method

Bath was prepared with the following recipe:
Dye -20 g/l, Cross-linking agent (DMDHEU) -100g/l, Basosoft EUK (Softening agent) -25 g/l, Magnesium Chloride -10 g/l, and Citric acid -5 g/l. pH (5-6) level was maintained by adding citric or acetic acid. The fabric was padded with the above mixed solution to give liquor pick up 100%. The fabric was dried and cured at 120-160 ^{o}C for 2-3 minutes.

Preparation of Cross-linking Solution with Fixapret CPN

Padding bath was prepared with the following recipe:
Portion M dye -20 g/l, Fixapret CPN–X -100g/l, Basosot E-UK- 20 g/l, citric acid -5 g/l, and Catalyst ($MgCl_2$) -10 g/l . Firstly, the Procion M dyes were dissolved by pasting with cold water followed by pouring warm water. The required amount of predissolved dyes and other dissolved chemicals were mixed together. The required volume was prepared, and the pH of the solution was adjusted to 5-6.

Preparation of Cross-linking Solution with Indosol E-50 powder

The impregnation bath was prepared by maintaining the following recipe:

1. Procion M dye -20 g/l
2. Indosol E-50 powder -40 g/l, Catalyst ($MgCl_2$) -12 g/l, Basosoft EUK -20 g/l, and Acetic acid -3 g/l.

Application of Fixapret CPN to Jute fabrics (Dry Cross-linking Method)

The prepared mixed solution was poured to the padding bath of two bowl padding machine and bleached CBC was padded (impregnated) through the above formulations to yield a wet pick up 90-100 %. The fabrics were then dried, cured at 160 ^{o}C for 2–3 minutes in a stenter/heat setting machine, and soaped (with alkali and detergents) for 10 minutes at 75 ^{o}C in order to remove the unfixed dyes. In brief, the sequence of operation was as follows: Padding > drying > curing > washing > drying.

Application of Indosol E-50 Powder with Reactive (Dry Cross-linking Method)

Firstly, fabric was padded with the predissolved dye solution (solution A). Then it was padded in Indosol E-50 powder solution (Solution B) to yield a wet pick up 90-100 %. The fabrics were then dried, cured at different temperatures like 120, 130, 140, 150, and 160^{o}C for 2-3 minutes in a stenter/heat setting machine, and soaped (with alkali and detergents) for 10 minutes at 75^{o}C in order to remove the unfixed dyes.

Sequences of Operation

Padding (in solution 'A') > Padding (in solution'B') > Drying > Curing

Testing

Dye fixation of various cross-linked dyed fabrics, fastness properties like washing, rubbing, light, and perspiration were determined by ASTM Standard Methods D 2096-11. Tensile strength of the fabrics was also determined by ASTM Standard Methods D 5035.

Testing the Cross-linking Reaction

The reactant fixation with cross-linking agents (Fixapret CPN, Fixapret ECOs and Indosol E-50 Powder) can be tested with dimethyl formamide boiling off test. The extract should not be more than very slightly tinted when fixation is completed. The dyed samples were boiled for 2 hours in a dimethyl formamide/water solution 1:1 with reflux condenser. In contrast to an unfixed dyeing, the cross-linked dyeing was not stripped by this method.

Results and Discussion

The dye fixation, fastness properties, and tensile strength of the cross linked (Fixapret CPN, Indosol E-50) dyed fabrics are shown in the following tables (1-7).

Effect of Cross-linking Agents and Temperature on Tensile Strength

It has been observed from Table 1 that tensile strength of the fabric decreases with the increase of the concentration of cross-linking agent as well as increase of curing temperature. In comparison to other cross-linking agents, Indosol E-50 powder caused the least (10%) loss in the strength of fabric. Hence, Indosol E-50 powder was found as an effective cross-linking agent for the promotion of dye fixation and breaking strength in simultaneous dyeing and finishing of jute fabric at concentration of 40g/l. Fixapret CPN may also be used where the tensile strength of the fabric is not so important.

***Table 1**. Effect of Cross-linking agents on Tensile Strength at different concentrations curing at different temperatures*

Name of Cross-linking agents	Concentration (G/l)	Tensile Strength (in kg) at different curing temperatures					
		110ºC	120ºC	130ºC	140ºC	150ºC	160ºC
Fixapret CPN	80	47.0	45.0	40.5	40.0	38.5	38.00
	90	46.5	44.0	40.0	37.5	35.0	30.50
	100	45.0	42.0	38.5	35.3	32.0	30.00
	110	44.5	40.0	38.0	30.0	29.0	26.0
Indosol E-50 Powder	20	48.0	48.5	48.0	47.0	46.0	44.0
	30	48.5	48.0	47.5	47.0	45.0	43.0
	40	48.0	47.5	46.5	45.0	44.5	42.0
	50	47.0	47.0	46.0	45.5	45.0	40.0

Tensile Strength of white fabric = 50 kg
Dye = Procion Orange MX2R = 20 g/l, Catalyst = $MgCl_2$, 6 H_2O = 12 g/l
Softener = Basosoft EUK = 30 g/l, Curing Time = 2 min.

Dye Fixation of Various Dyed Fabrics Cross-linked with Fixapret CPN and Indosol E-50 Powder

Seven kinds of Procion M (ICI) types of reactive dyes were selected and applied to jute fabrics with Fixapret CPN (BASF) and Indosol E-50 powder (Sandoz) in simultaneous dyeing and finishing by dry cross-linking method. Dye fixation of the dyed fabrics was measured and given in Table 2 and Table 3. The results in Table 2 showed that the fabrics dyed with various reactive dyes cross-linked with Fixapret CPN and obtained dye fixation ranges from 60-70%. On the other hand, the dyed fabric cross-linked with Indosol E-50 powder obtained (Table 3) dye fixation ranges from 92-95%, which is highly satisfactory. This high fixation occurred due to stable chemical bond formed with both dye and fiber in the presence of catalyst by cross-linking technique. More so, the color will not be washed out even after prolonged boiling with dimethyl formamide solution.

***Table 2**. Dye fixation of various dyed fabrics cross-linked with Fixapret CPN*

Name of dyes	CI Generic Name	Dye fixation (%)
Procion Yellow MX4R	CI Reactive Orange 14	70
Procion Red MX8B	CI Reactive Red 11	67
Procion Orange MX2R	CI Reactive Orange 4	70
Procion Brown MXGRN	CI Reactive Brown 23	65
Procion Blue MX2R	CI Reactive Blue 140	60
Procion Yellow MX3G	CI Reactive Yellow 7	68
Procion Turquoise MXG	CI Reactive Blue 140	65

Fixapret CPN = 100g/l, $MgCl_2$, $6H_2O$ = 12 g/l, Curing temp. = 160 ºC,
Time = 2 min.

Table 3. *Dye fixation of cross-linked dyed fabrics using various reactive dyes and Indosol E-50 powder (optimum condition)*

Name of dyes	C.I Generic name	Dye fixation (%)
Procion yellow M4R	CI Reactive Orange 14	93
Procion Red M8B	CI Reactive Red II	94
Procion Orange Mx2R	CI Reactive Orange 4	94
Procion Brown MxGRN	CI Reactive Brown 23	92
Procion Blue Mx2R	CI Reactive Blue 4	92
Procion Turquoise MxG	CI Reactive Blue 140	95
Procion Yellow Mx3G	CI Reactive Yellow 7	95

Cross-linking agent, Indosol E-50 powder = 40g/l, Curing temp. =140 °C,Curing time = 2 min

Fastness Properties of Cross-linked Dyed Fabrics of Various Reactive Dyes

Seven varieties of Procion M (ICI) types of reactive dyes were selected and applied to jute fabrics. This was done by cross-linking dyeing method using Fixapret CPN (BASF) and Indosol E-50 Powder as cross-linking agent. Fastness properties such as washing, staining, rubbing, light, and perspiration were determined and assessed against blue scale (1-5) and blue standards (1-8). The results are shown in Table 4, Table 5, and Table 6. The washing, staining, rubbing, and light fastness of the cross-linked dyed fabrics are independent of the nature of dye. In most cases, the washing and rubbing fastness were of high standard figuring from 4-5 and light fastness was fair to good rating from 4-5. This is seen in Table 4 and Table 5 where international highest figure is 8. There was no marked change in the case of Fixapret CPN and Indosol E-50 powder.

Table 4. *Fastness Properties of the cross-linked dyed fabrics using Fixapret CPN*

Name of dyes	Fastness properties			
	Washing (ISO-3)	Staining on cotton	Rubbing	Light
Procion Yellow MX4R	4-5	4	5	5
Procion Red MX8B	4	4	4-5	4-5
Procion Orange MX2R	4	4	5	5
Procion Brown MXGRN	4-5	5	5	5
Procion Blue MX2R	4	5	4	4-5
Procion Yellow MX3G	3-4	4	4	4
Procion Turquoise MXG	4-5	5	5	4-5

Fixapret CPN = 100 g/l, $MgCl_2$, $6H_2O$ = 12 g/l, Curing Temp. = 160 °C, Time = 2 min

***Table 5**. Fastness properties of the cross-linked dyed fabric using various reactive dyes and Indosol E-50 Powder*

Name of dyes	CI Generic Name	Fastness Properties		
		Washing (ISO-3)	Rubbing	Light
Procion yellow MX4R	Reactive Orange 14	5	5	4-5
Procion Red M8B	Reactive Red 112	4-5	5	4
Procion Orange MX2R	Reactive Orange 4	5	5	4-5
Procion Brown MXGRN	Reactive Brown 23	4	5	4-5
Procion Blue MX2R	Reactive Blue 4	5	4-5	5
Procion Yellow MX3G	Reactive Yellow 7	4-5	5	4-5
Procion Turquoise MXG	Reactive Blue 140	4	4-5	4-5

Indosol E-50 Powder = 40 g/l, Curing temp. = 140 ^{o}C, curing time = 2 min.

In Table 6 and Table 7, fastness to perspiration (acid and alkali) was very good figuring from 3 to 4 for Fixapret CPN and 4 to 5 for Indosol E-50 powder. The cross-linked method produced bright shade of color. Needless to say, all these properties are satisfactory.

***Table 6.** Fastness to perspiration (Acid and Alkali) of cross-linked dyed fabrics using Fixapret CPN*

Name of dyes	Acid Perspiration		Alkali Perspiration	
	Change of Shade	Staining	Change of shade	Staining
Procion Yellow MX 4R	3	4	4	3-4
Procion Red MX8B	4	4	3-4	4
Procion Orange MX2R	4	3	3-4	4
Procion Brown MX GRN	4	4	4	4
Procion Blue M X 2R	3-4	4	4	4
Procion Turquoise MXG	4	4	4	4
Procion Yellow M X 3 G	4	4-5	3-4	4

***Table 7.** Fastness to perspiration (Acid and Alkali) of cross-linked dyed fabrics using Indosol E-50 powder*

Name of dyes	Acid Perspiration		Alkali Perspiration	
	Change of Shade	Staining	Change of shade	Staining
Procion Yellow MX 4R	4	5	4	4
Procion Red MX8B	4	4	5	4-5
Procion Orange MX2R	4-5	5	4	4
Procion Brown MX GRN	4	4	4	4
Procion Blue M X 2R	4-5	4	5	4-5
Procion Turquoise MXG	4	4	3-4	4
Procion Yellow M X 3 G	5	5	4-5	4

Conclusion

Bleached jute fabrics dyed and finished simultaneously with various dyes and Indosol E-50 powder (Sandoz) applying pad-dry-cure method showed excellent performance in respect of all dyeing properties. Fixapret CPN (DMDHEU) of BASF achieved almost satisfactory results, whereas

Fixapret ECOs attained unexpected results as it did not react with dyes. The fastness to washing, staining, rubbing, and perspiration of the cross-linked dyed fabrics using various reactive dyes was found to be excellent. The color shade produced in the developed dyeing method is brilliant and uniform as the color penetrates well.

When Fixapret CPN (DMDHEU) is used in this technique, significant loss in tensile strength of fabric occurs. However, these fabrics may be used for the purposes where tensile strength property is not so important.

This is a new cross-linking dyeing method with high dye fixation, low dye cost, simple application, high fastness rating, improved crease recovery with less loss of tensile strength without any shrinkage, brilliant shade, increased productivity, savings in labor, energy, water & time, and low effluent pollution.

In this method, continuous production is possible without the installation of special equipment in plants where resin finishing is normally done.

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